

Hardness as CaCO₃ by EDTA Titrimetric**SM 2340 C – 1997 (2011)**

ADDITIONAL QC REQUIREMENTS FOR THIS METHOD: Certified or Accredited laboratories using this method are assessed to applicable requirements of SM 1020 and SM 2020.

Facility Name: _____ VELAP ID: _____

Assessor Name: _____ Analyst Name: _____ Inspection Date: _____

Records Examined: SOP Number/ Revision/ Date: _____ Analyst: _____

Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
1) Are samples preserved with HNO ₃ or H ₂ SO ₄ to pH<2 and analyzed within 6 months?	40CFR 136.3 Table 11				
2) Are wastewater samples pretreated by nitric acid-sulfuric acid or nitric acid- perchloric acid digestion as described in section 3030- 1997 (2004)?	3.a				
3) If digesting samples using nitric acid-sulfuric acid (3030G), are the following steps followed? <input type="checkbox"/> Add 5 mL of HNO ₃ to sample, boil sample, and evaporate to 15-20 mL. <input type="checkbox"/> Add 5 mL of HNO ₃ and 10 mL of H ₂ SO ₄ , and evaporate until dense white fumes just appear. <input type="checkbox"/> If solution does not clear, add 10 mL HNO ₃ and repeat until solution is clear and no brownish fumes. <input type="checkbox"/> Cool, dilute to ~50 mL, and boil to dissolve salts. <input type="checkbox"/> Cool, rinse into a volumetric flask and dilute to 100 mL.	3030G, 3030E				
4) If digesting samples using nitric acid- perchloric acid, are the following steps followed? <input type="checkbox"/> Add 5 mL HNO ₃ to sample, evaporate to 15-20 mL. <input type="checkbox"/> Add 10 mL HNO ₃ and 10 mL HClO ₄ , and evaporate until dense white fumes just appear. <input type="checkbox"/> If solution does not clear, keep boiling until clear. <input type="checkbox"/> Cool, dilute to ~50 mL, and boil to remove chlorine or oxides of nitrogen. <input type="checkbox"/> Cool, rinse into a volumetric flask and dilute to 100 mL.	3030H, 3030E				
5) Were titrations performed at or near “normal room temperature”?	1.c				
6) Is a sample volume selected that requires less than 15 mL EDTA titrant?	3.b				
7) Is a sample aliquot of 25 mL (unless less than 5 mg/L hardness) diluted to 50 mL with distilled water, and 1 to 2 mL buffer added?	3.b, 3.c				

Notes/ Comments:

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8) For samples with hardness less than 5 mg/L, is a large aliquot of 100 to 1000 mL titrated, using proportionally larger amounts of buffer, inhibitor, and indicator?	3.c				
9) Following the addition of buffer, is an appropriate amount of indicator added per 50 mL sample? <input type="checkbox"/> 2 drops Eriochrome Black T solution <input type="checkbox"/> 1 mL Calgamite solution <input type="checkbox"/> minimal amount of commercially prepared dry-powder form of either Eriochrome Black T or Calgamite	2.c, 3.b				
10) Following the addition of indicator, is EDTA titrant added slowly, with continuous stirring, until the last reddish tinge disappears? <u>Notes:</u> * <i>Solution normally turns blue at the end point.</i> * <i>Daylight or a daylight fluorescent lamp is recommended because incandescent lights tend to produce a reddish tinge in the blue at the endpoint.</i>	3.b				
11) Is titration completed within 5 minutes, measured from time of buffer addition?	3.b				
12) Is the buffer solution discarded when 1 or 2 mL added to the sample fails to produce a pH of 10.0 ± 0.1 at the titration end point?	2.a.2				
13) For samples with hardness less than 5 mg/L, is the volume of EDTA used for the blank subtracted from the volume of EDTA used for the sample?	3.c				
14) Is hardness calculated using the following formula? Hardness, mg/L = (A x B x 1000) / mL sample Where A = mL titration for sample B = mg CaCO ₃ equivalent to 1.00 mL EDTA titrant	4				

Notes/Comments: